

AD 678395

FTD-HT-23-1252-67

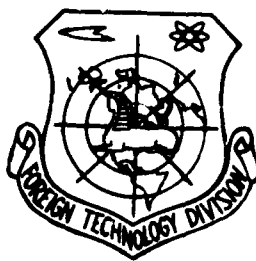
FOREIGN TECHNOLOGY DIVISION



STUDY OF ADDITIVES SYNTHESIZED FROM DERIVATIVES OF NAPHTHENIC HYDROCARBONS

by

I. M. Orudzheva and Sh. M. Novruzov



DEC 1 1968

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EDITED TRANSLATION

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English pages: 4

SOURCE: Azerbaydzhanskoye Neftyanoye Khozyaystvo
(Azerbaydzhan Petroleum Economy), 1965,
pp 37-38.

Translated by: F. Dion/TDBRO-2

TT7002329

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PREPARED BY:

TRANSLATION DIVISION
FOREIGN TECHNOLOGY DIVISION
WP-APB, ONO.

DATA HANDLING PAGE				
61-ACCESSION NO. TT7002329	62-DOCUMENT LOC	63-TOPIC TAGS antioxidant additive, lubricating oil		
64-TITLE STUDY OF ADDITIVES SYNTHESIZED FROM DERIVATIVES OF NAPHTHENIC HYDROCARBONS				
65-SUBJECT AREA 11				
66-AUTHOR/CO-AUTHORS ORUDZHEVA, I. M.; NOVRUZOV, SH. M.			67-DATE OF INFO -----65	
68-SOURCE AZERBAYDZHANSKOYE NEFTYANOYE KHOZYAYSTVO (RUSSIAN)			69-DOCUMENT NO. FTD-HT-23-1252-67	
			70-PROJECT NO. 72301-78	
71-SECURITY AND DOWNGRADING INFORMATION UNCL, 0			72-CONTROL MARKINGS NONE	73-HEADER CLASN UNCL
74-REEL/FRA ME NO. 1883 0701	75-SUPERSEDES	76-CHANGES	77-GEOGRAPHICAL AREA UR	78-NO. OF PAGES 4
79-CONTRACT NO.	80-REF ACC. NO. AR6024954	81-PUBLISHING DATE	82-TYPE PRODUCT Translation	83-REVISION FREQ None
84-STEP NO. UR/0000/65/000/000/0037/0038			85-ACCESSION NO. TT7002329	
<p>ABSTRACT</p> <p>In order to develop antioxidant motor-oil additives whose composition includes phosphorus and sulfur, monochlorides of cyclohexyl- and methylcyclohexylphosphorous acids were prepared. The chlorides obtained were subjected to condensation reactions with various alkyl phenols (C_3-C_{10}) and sulfide-disulfide alkyl phenols. The effect of the synthesized compounds on the performance characteristics of D-11 oil was investigated. Tests of these compounds for the oxidation resistance, thermal stability, and anticorrosive properties of the diesel oil showed that the induction period of the oxidation, determined by the method of the AzNII, increases from 30 to 70-100 min, and the time of absorption of 10 ml of oxygen, from 240 to 340-355 min. The anticorrosive properties were tested under drastic conditions (method of NAMI); in the presence of the additives, the corrosion decreases from 360 to 1-13 g/m². The thermal stability of D-11 oil increases from 25 to 60-105 min. Phosphorus- and sulfur-containing compounds in compounds in combination with SB-3 and BFK additives have practically no effect on the wetting properties of the oils.</p>				

STUDY OF ADDITIVES SYNTHESIZED FROM DERIVATIVES OF NAPHTHENIC HYDROCARBONS

I. M. Orudzheva and Sh. M. Novruzov

To obtain antioxidant additives to motor oils we synthesized P- and SP-containing compounds. Cyclohexanol and methylcyclohexanol were taken as the initial raw materials. The reaction of these alcohols with phosphorus trichloride produced the corresponding monochlorides of cyclohexyl and methylcyclohexyl phosphorous acids.

The synthesized acid chlorides were subjected to condensation with various alkyl phenols (C_3-C_{10}), and as a result a series of triply substituted phosphites was obtained.

These phosphites contain 5.8–9.2% phosphorus. We obtained P-containing additives, for which the monochlorides of dicyclohexyl phosphorous acid were reacted with sulfide-disulfide alkyl phenols. The characteristics of the obtained P- and PS- containing additives are given in Table 1.

We studied the effect of these compounds on the performance characteristics of D-11 oil. Mixed with the new additives, D-11 oil was tested for stability, thermal stability and anticorrosion properties.

The results of these tests are given in Tables 2 and 3.

From the data of Table 2 it is evident that in small concentrations (about 0.1%) the phosphites very effectively improve the stability of oils (as determined by the AzNII method) at 175°C. The induction period of oxidation increases from 30 to 70–100 min, and the time for absorption of 10 ml of oxygen, from 240 to 340–355 min.

All the synthesized compounds increase the stability of D-11 oil identically. An analogous picture is observed in the tests of these for anticorrosion properties and thermal stability (Table 3).

Table 1. Elementary Composition of Compounds Synthesized from Naphthenic Hydrocarbons

Proposed Formula	Content, %			
	Carbon	Hydrogen	Phosphorous	Sulfur
$(C_6H_{11}O-)_2POC_6H_4C_3H_7$	69.37	8.95	9.20	—
$(CH_3C_6H_{10}O-)_2POC_6H_4C_3H_7$	70.68	9.83	7.56	—
$(CH_3C_6H_{10}O-)_2POC_6H_4C_4H_9$	70.48	9.23	7.89	—
$(C_6H_{11}O-)_2POC_6H_4C_5H_{11}$	70.62	9.54	7.90	—
$(C_6H_{11}O-)_2POC_6H_4C_{10}H_{21}$	73.02	10.36	5.84	—
$(C_6H_{11}O-)_2P \quad P(-OC_6H_{11})_2$ $\quad \quad \quad O \quad \quad O$ $H_7C_3-C_6H_3-S-C_6H_3-C_3H_7$	66.91	7.90	8.54	4.83
$(C_6H_{11}O-)_2P \quad P(-OC_6H_{11})_2$ $\quad \quad \quad O \quad \quad O$ $H_9C_4-C_6H_3-S-C_6H_3-C_4H_9$	66.79	8.10	7.32	3.94
$(C_6H_{11}O-)_2P \quad P(-OC_6H_{11})_2$ $\quad \quad \quad O \quad \quad O$ $H_{11}C_5-C_6H_3-S-C_6H_3-C_5H_{11}$	66.97	9.42	6.90	3.51
$(C_6H_{11}O-)_2P \quad P(-OC_6H_{11})_2$ $\quad \quad \quad O \quad \quad O$ $H_9C_4-C_6H_3-S_2-C_6H_3-C_4H_9$	64.86	8.50	8.06	6.94
$(C_6H_{11}O-)_2P \quad P(-OC_6H_{11})_2$ $\quad \quad \quad O \quad \quad O$ $H_{11}C_5-C_6H_3-S_2-C_6H_3-C_5H_{11}$	65.83	8.00	7.69	7.00

Table 2. Stability of D-11 Oil Mixed with Different Antioxidant Additives by the AzNII Method

Sample	Quantity of Additive, %	Induction Period, min	Time for Absorption of 10 ml of Oxygen, min
Oil Without Additive	—	30	240
Oil + Additive No. 1	0.1	63	335
Oil + Additive No. 2	0.05	93	354
Oil + Additive No. 2	0.3	95	355
Oil + Additive No. 3	0.1	108	341
Oil + Additive No. 4	0.1	77	341
Oil + Additive No. 5	0.1	76	354
Oil + Additive No. 5	0.05	61	321

Table 3. Effect of P- and S-Containing Compounds on the Anticorrosion and Thermal-Stability Properties

Sample	Quantity of Additive, %	Thermal oxidation resistance by the Papok Method, min	Coef. of laquer deposition, K ₁	Laquer residue, %	Corrosion by the NAMI Method, 25 h, g/m ²
Oil Without Additive	—	25	0.89	22	360
Oil + Additive No. 1	0.5	50	0.19	9.5	—
Oil + Additive No. 1	1.0	98	0.08	8	27.0
Oil + Additive No. 4	0.5	57.5	0.24	14	1.65
Oil + Additive No. 4	1.0	105	0.05	6	4.65
Oil + Additive No. 5	0.5	54.2	0.25	14	5.0
Oil + Additive No. 5	1.0	94	0.09	9	8.7
Oil + Additive No. 6	1.0	74	0.13	13	6.35
Oil + Additive No. 7	1.0	75	0.12	12	6.0
Oil + Additive No. 8	1.0	73	0.14	10.5	14
Oil + Additive No. 9	1.0	53	0.17	9	3.9
Oil + Additive No. 10	1.0	49	0.18	9	8.4

Table 4. Wetting Properties of D-11 Oil with Additives by the PZV Method

Sample	Wetting Properties, Units
Oil Without Additive	5 to 5.5
Oil + 5% SB-3	1 to 1.5
Oil + 6% BFK	0.5 to 1.0
Oil + 5% SB-3 + 1% Additive No. 8	0.5
Oil + 6% BFK + 1% Additive No. 8	0.5
Oil + 6% BFK + 1% Additive No. 4	0 to 0.5

The anticorrosion properties of D-11 oil were tested under drastic conditions by the NAMI method for 25 h in the presence of a catalyst, copper petronate. When 0.5-1.0% of the P-containing compounds are added to D-11 oil corrosion of the oil is reduced from 360 to 1-13 g/m². The phosphites also effectively increase the thermal stability of D-11 oil and reduce the coefficient of laquer deposition (Table 3).

P- and S-containing compounds produced from cyclohexanol and containing 6.9-8.5% P and 3.5-6.9% S increase the thermal stability of D-11 oil and improve its anticorrosion properties (Table 3). In combination with SB-3 and BFK additives these compounds do not worsen the wetting properties of diesel oil (Table 4).

Thus, from derivatives of naphthenic hydrocarbons we synthesized a number of new S- and P-containing compounds that in pure form and in combination with other known multipurpose additives improve the performance characteristics of oils.